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(54) Title: METHOD FOR THE ONE STEP PREPARATION OF TEXTILES

(57) Abstract: A one step method for the treatment of a non-finished textile component is provided. The method comprises the combination of a bleaching and de-sizing treatment steps into a single step and comprises treating a non-finished textile component in an aqueous treatment solution of a hydrophobic bleaching system and a de-sizing system. Preferred hydrophobic bleaching systems are combinations of hydrogen peroxide and a hydrophobic bleach activator or a hydrophobic peracid. Preferred bleach activators include the alkanoyloxybenzene sulfonates and in particular noanoyloxybenzene sulfonate. The method provides superior fiber protection and fabric strength benefits versus conventional processing as well as allows significant savings in both energy and time due to the elimination of multiple washing and/or drying stages and lower bleaching temperatures.

## METHOD FOR THE ONE STEP PREPARATION OF TEXTILES

Cross Reference to Related Application

5 This application claims priority under 37 U.S.C. § 119(e) to U. S. Provisional Application  
Serial No.60/182,611, filed February 15, 2000 (Attorney Docket No.7959P).

Technical Field

The present invention relates to the one step preparation of textile components and, more particularly, to the simultaneous de-sizing and bleaching of textile components such as woven fabrics, fibers or yarns through the use of hydrophobically activated bleaching systems.

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Background of the Invention

In the textile processing of natural fibers, yarns and fabrics, a pretreatment or preparation step is typically required to properly prepare the natural materials for further use and in particular for the dyeing and/or finishing stages typically required for commercial goods. These textile treatment steps remove impurities and color bodies, either naturally existing or those added by the spinning and weaving steps to the fibers and/or fabrics.

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While textile treatments may include a number of varying treatments and stages, the most common include: singeing - the removal of loose or miscellaneous fibers from the surface by burning with a flame; de-sizing - the removal of sizing agents, such as starches, via enzymatic, alkali or oxidative soaking; scouring - the removal of greases, oils, waxes and fats by contact with a solution of sodium hydroxide at temperatures near boiling; mercerization - the application of high levels of sodium hydroxide in conjunction with stretching and pulling of the fabrics for increased fiber strength and bleaching - the use of hydrogen peroxide bleaching systems at temperatures near boiling for the removal of color bodies and whitening of fabrics.

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Commercial processing typically requires the separation of these pre-treatment steps due to the broad variation of conditions present in each of the steps. However, this separation of treatment steps leads to heavy additional costs added to the overall treatment process due to the requirement of multiple rinsing and/or drying steps required between the respective stages. The additional rinse and/or drying steps add enormous addition costs and waste materials to the treatment process.

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Accordingly, the combination of various pre-treatment stages into a one-step treatment would have a significant impact in the commercial treatment of textiles in the form of reduced costs and waste materials over the commercial processes typically employed. Three treatment

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stages which are commonly investigated for combination into a one-step process are de-sizing, scouring and bleaching.

However, the combination of these three common steps has previously been unsatisfactory. Currently employed bleaching technology involves the use of hydrogen peroxide bleaching at temperatures in excess of 95 °C. Such high temperatures and strong bleaching systems are wholly incompatible with the amylase enzymes necessary in a de-sizing operation. Thus, the combination of the de-sizing and bleaching technology at temperatures in excess of 95 °C leads to destruction of the de-sizing enzymes and a unsatisfactory de-sizing result. Alternative de-sizing techniques such as alkali or oxidative soaking involves the use of aggressive chemicals which lead to fiber damage. On the other hand, reduction of the temperature at which the one-step treatment is conducted to allow effective enzymatic de-sizing results in an unacceptably poor bleaching with whiteness values below the commercially acceptable limit.

Lower temperature bleaching systems have been investigated in textile processing to little commercial success. Hydrophilic activation of the hydrogen peroxide has been attempted. In particular, tetra acetyl ethylene diamine or TAED is a common hydrophilic bleach activator widely accepted in the consumer laundry bleaching applications to provide effective bleaching at lower wash temperatures. TAED has been taught in hydrogen peroxide textile bleaching, and in particular in the bleaching of regenerated cellulose such as rayon. However, while TAED has allowed lower bleaching temperatures, it has proven to provide little advantage in the fabric strength reduction which results from high temperature bleaching and has not provided the satisfactory whiteness profiles necessary for commercial goods. In addition, the poor water solubility of TAED limits its application in textile processing.

Hydrophobic bleach activators, such as nonanoyloxybenzene sulfonate, sodium salt (NOBS) have been employed in consumer laundry detergent applications such as Tide® with Bleach to work in conjunction with peroxygen sources to provide activated bleaching in consumer laundering of garments. Activated bleaching in consumer home laundry conditions allows effective cleaning of certain soils and stains in cold water temperatures. The use of hydrophobic systems has been limited in a home consumer laundry environment due to the formation of diacyl peroxides in the wash solution. Diacyl peroxides degrade natural rubber components into which they come in contact. Thus, sump hoses, rubber gaskets, etc in laundry machines have prevented exploitation of this technology in certain geographies.

The severe conditions employed in the bleaching of textiles have heretofore prevented the successful application of laundry detergent bleaching technology in textile mill applications. Indeed, EP 584,710 discloses the use of activated bleaching in textile mill applications wherein

NOBS is briefly disclosed along with a multitude of other classes and types of activators. While NOBS is disclosed, there is no successful application of hydrophobic bleaching technology where acceptable whiteness values are achieved while damage to fabrics and fibers is minimized. Indeed, EP 584,710 specifies that in order to achieve acceptable whiteness benefits, additional alkali bleaching is necessary which will dramatically increase fiber damage.

Accordingly, the need remains for an effective one step textile treatment process and in particular for the combination of de-sizing, scouring and bleaching in textile treatment which can provide superior wettability and whiteness benefits while providing improved fabric strength retention and reduced fiber damage versus conventional textile bleaching processes.

#### Summary of the Invention

This need is met by the present invention wherein a one step or simultaneous treatment method for textiles and superior performing fabrics obtained therefrom is provided. The method of the present invention provides textiles with commercially acceptable whiteness profiles and superior fabric strength retention and fiber damage properties to fibers processed via the method. The method of the present invention involves the combination of de-sizing and a peroxide based bleaching system via the use of hydrogen peroxide and a hydrophobic bleaching agent such as an activator or a hydrophobic peracid. . In addition, the method of the present invention allows for significant cost reduction in the operation of a textile bleaching process through the one step treatment of textiles and the use of significantly lower bleaching temperatures than conventional peroxide bleaching and much shorter processing times, particularly in batch processing.

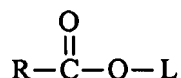
While not wishing to be bound by theory, it is believed that the hydrophobic bleach species of the present invention provide better absorbency on the fabrics and yarns and better "wetting" of the surface of the fibers than conventional peroxide bleaching techniques or hydrophilic activators. Hydrophobic bleach activators form the active bleaching species, peracid, on the surface of the fabric allowing a longer time on the surface of the fabric. Hydrophilic activators, meanwhile, form peracid in solution and must then undergo a fabric solution interaction which is less efficient. As a result, the hydrophobic activators of the present invention provide superior bleaching and whiteness while minimizing fiber damage and strength reduction.

According to a first embodiment of the present invention, a method for the simultaneous treatment of non-finished textile components is provided. The method comprises the steps of providing a non-finished textile component such as a fiber, yarn or fabric, saturating the textile component with an aqueous treatment solution comprising a hydrophobic bleaching system and a

de-sizing system and allowing the treatment solution to remain in contact with the textile component for a period of time sufficient to treat the textile component.

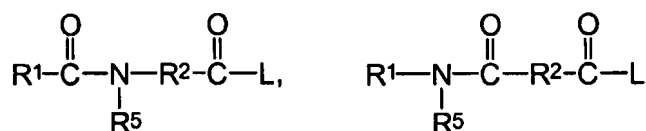
Preferably, the treatment solution is the combination of hydrogen peroxide and a hydrophobic bleaching agent such as an activator or a pre-formed hydrophobic peracid with the combination of hydrogen peroxide and a hydrophobic bleach activator being the most preferred. The bleach activator, when present, is selected from the group consisting of :

a) a bleach activator of the general formula:



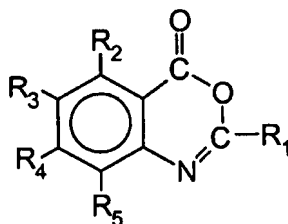
wherein R is an alkyl group having from about 5 to about 17, preferably from about 7 to about 11, carbon atoms and L is a leaving group;

b) a bleach activator of the general formula:



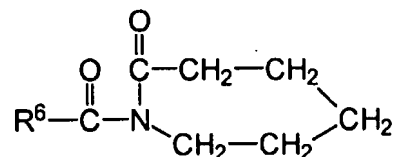
or mixtures thereof, wherein R<sup>1</sup> is an alkyl, aryl, or alkaryl group containing from about 1 to about 14 carbon atoms, R<sup>2</sup> is an alkylene, arylene or alkarylene group containing from about 1 to about 14 carbon atoms, R<sup>5</sup> is H or an alkyl, aryl, or alkaryl group containing from about 1 to about 10 carbon atoms, and L is a leaving group;

c) a benzoxazin-type bleach activator of the formula:



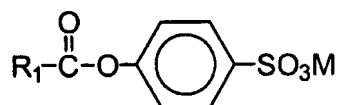
wherein R<sub>1</sub> is H, alkyl, alkaryl, aryl, arylalkyl, and wherein R<sub>2</sub>, R<sub>3</sub>, R<sub>4</sub>, and R<sub>5</sub> may be the same or different substituents selected from H, halogen, alkyl, alkenyl, aryl, hydroxyl, alkoxy, amino, alkylamino, -COOR<sub>6</sub>, wherein R<sub>6</sub> is H or an alkyl group and carbonyl functions;

d) a N-acyl caprolactam bleach activator of the formula:



wherein  $\text{R}^6$  is H or an alkyl, aryl, alkoxyaryl, or alkaryl group containing from 1 to 12 carbons; and

e) mixtures of a, b, c and d with the alkanoyloxybenzenesulfonates of the formula:



wherein  $\text{R}_1$  is an alkyl group having from about 7 to 11 carbon atoms and M is a suitable cation being the most preferred hydrophobic activators. Preferred de-sizing agents are enzymatic de-sizing.

In optional embodiments, the treatment solution further includes an ingredient selected from the group consisting of wetting agents, sequestering agents, stabilizing agents, scouring agents and mixtures thereof. Preferred ranges of peroxide and activator, when present, are from about 1g/L to about 50g/L hydrogen peroxide with a molar ratio of hydrophobic bleach activator to hydrogen peroxide of from about 1:1 to 1:50.

The textile components remain in contact with the treatment solution at a temperature of from about 20 to about 80 °C, preferably from about 50°C to about 80 °C and for a period of time suitable for treating the textile component which is typically at least about 15 minutes, more typically from about 30 minutes to about 180 minutes, even more typically from about 30 to about 120 minutes and most typically from about 30 to about 90 minutes. In preferred operations, the textile component experiences a fabric strength reduction of less than about 10% and more preferably less than about 5% due to the bleaching of the present invention. Alternatively, the textile component experiences a fluidity increase of less than about 25%.

In accordance to a second embodiment of the present invention, a method for the one step batch treatment of a non-finished textile woven fabric is provided. This method comprises the steps of:

- a) providing an incoming non-finished woven fabric;
- b) passing the woven fabric to an aqueous treatment solution comprising a hydrophobic bleaching system and a de-sizing system, heating the treatment solution to a temperature of from about 20 to about 80 °C and allowing the bleaching solution to

contact the fabric for a period of time of from about 15 to about 180 minutes, wherein the resultant treated textile components have a whiteness on the CIE scale of at least about 70.

In accordance with yet another embodiment of the present invention, a method for the one  
5 step preparation of a non-finished textile component is provided. The method comprises the steps of providing a non-finished textile component, saturating the textile component with an aqueous treatment solution comprising a) a hydrophobic bleaching system of hydrogen peroxide and a hydrophobic bleach activator; b) a de-sizing system and c) a alkaline scouring agent; and allowing the treatment solution to remain in contact with the textile component for a period of time  
10 sufficient to treat the textile component.

Accordingly, it is an object of the present invention to provide a method for the one step treatment of textile components such as fibers, yarns and fabrics which provides superior whiteness on textiles without a significant reduction in fabric strength, and potential fiber damage common to conventional preparation processes. It is another object of the present invention to  
15 provide a one step method for the treatment of textile components employing a hydrophobic bleach activator or pre-formed peracid in conjunction with hydrogen peroxide. These, and other objects, features and advantages of the present invention will be apparent from the following detailed description and the appended claims.

All percentages, ratios and proportions herein are on a 100% weight basis unless  
20 otherwise indicated. All documents cited herein are hereby incorporated by reference

#### Detailed Description of the Preferred Embodiments

According to the present invention, a superior textile treatment process for fibers, yarns and fabrics, particularly on woven goods, is provided. The proper preparation of a textile  
25 component such as a fiber, yarn or fabric is critical to the success of further treatment in the manufacture of commercially feasible textile components such as yarns, fabrics, garments, and the like. These treatment steps include dyeing, printing and/or finishing such as application of durable press finishes. Uneven color appearance or impurities such as waxes or oils on the surface of the textile prevent the uniform application of many treatments. Present commercial  
30 textile preparation methods, and, in particular, textile bleaching methods, remain unsatisfactory due to the fiber and fabric damage of the treated textiles, high costs associated with the high temperatures necessary to drive bleaching, high costs due to extra equipment necessary for separate treatment steps for de-sizing, scouring and bleaching, and environmental unfriendliness. .

The present invention provides a cost effective and superior performing alternative to conventional processing. The present invention involves a one step or simultaneous treatment of textiles wherein bleaching and de-sizing are combined into a single treatment step via the use of a hydrophobic bleaching system and a de-sizing system which is preferably an enzymatic de-sizing system. Optionally, an alkaline scouring step may also be combined with the treatment bath thereby providing a one step treatment for all three key textile treatment steps. This simultaneous treatment is made possible via the use of a hydrophobic bleaching system such as the combination of a hydrophobic activator and hydrogen peroxide or a pre-formed hydrophobic peracid. These hydrophobic bleaching species provide superior results in the context of textile whiteness and in fabric strength retention at lower treatment temperatures which makes possible the use of enzymatic de-sizing agents in the same treatment solution and under the same conditions.

While conventional textile bleaching processes require high temperatures of more than 95 °C to achieve satisfactory whiteness values, the result is a degradation of the strength of the fabrics from the original fabric strength and a temperature at which enzymatic de-sizing agents are destroyed. Degradation of more than 15% of original fabric strength is not uncommon. On the other hand, lower bleaching temperatures at which enzymatic de-sizing agents will provide a useful result, i.e. less than about 80 °C, result in wettability and whiteness values well below satisfactory levels. Thus, it is the singular achievement of the method of the present invention which provides satisfactory de-sizing and whiteness values at temperatures at which enzymatic de-sizing agents remain productive.

Via the method of the present invention it is possible to achieve whiteness values of at least about 70 on the CIE whiteness index. In addition, via the method of the present invention, it is possible to deliver superior fabric strength retention benefits by providing a fabric strength reduction of less than about 10%, more preferably less than about 5% and most preferably less than about 3% from the original fabric strength. The method of the present invention also provides for superior fiber protection as opposed to conventional bleaching processes. Conventional textile bleaching leads to excessive fiber damage as measured by an increase in chemical degradation of more than 50% in typical applications. The method of the present invention, on the other hand, provides for chemical degradation in treated fibers of no more than 25%, preferably no more than 15% and even more preferably of no more than 10% whereby an increase in chemical degradation represents an increase in fiber damage. Accordingly, the use of the method of the present invention results in a significant reduction in fiber damage as opposed to conventional bleaching technology of peroxide at more than 95° which produces significantly higher fluidity increases. The unique collection of benefits of the present invention is achieved



through the identification of the effectiveness of hydrophobic bleaching species in the textile environment and is accomplished due to the lower bleaching temperatures and surface activity possible through these hydrophobic bleaching species.

In addition, the method of the present invention provides a significant cost advantage  
5 through the use of lower bleaching temperatures and the elimination of multiple rinsing and/or drying cycles that are required in conventional multi-step processing. In particular, the cost savings in both energy and time in batch type processing via the present invention is significant.

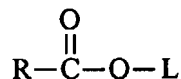
Conventional processing involves the immersion of the non-finished fabrics into a de-sizing bath, heating the de-sizing bath to an effective temperature of about 60 to about 70 °C  
10 followed by multiple rinsing steps, followed by immersion in a scouring bath, heating to an effective temperature and multiple rinsing steps followed by immersion in a bleaching solution and heating of the bleaching solution to the effective temperature of often more than 95 °C followed by multiple rinsing steps. The heating of the fabric and solution to such high temperatures involves a significant investment of both money and time for heating such large  
15 quantities of fabric and aqueous solution. In addition, the multiple rinsing steps generates enormous amounts of waste which must be treated thereby adding additional costs. However, due to the lower processing temperatures possible via the present invention and the effective combination of steps the present invention requires significantly less energy and time investment for heating and uses significantly less wash water thereby generating less waste and importantly  
20 saves considerable expense on necessary equipment and floor space in production facilities.

The term "simultaneously" is intended to indicate that at least a portion of the desizing and bleaching are carried out in a single operation. This has the obvious advantage that the washing and other treatments normally performed between separately conducted desizing and bleaching steps are no longer required. Thereby, the water and energy demand as well as the  
25 demand to different equipment to be used for each of the processes are considerably reduced. Furthermore, depending on the type of fabric to be treated and the nature of impurities present thereon, a scouring effect may be obtained during the performance of the process of the invention. Thus, in such cases, no additional scouring treatment need to be performed. While it is preferred that all de-sizing be carried out in conjunction with the bleaching step, one of ordinary skill in the  
30 art will recognize that some portion of de-sizing may be carried out separately from the bleaching step without departing from the spirit of the invention.

The present invention involves the use of a hydrophobic bleaching system which comprises either the combination of hydrogen peroxide and a hydrophobic bleach activator or the use of a pre-formed hydrophobic peracid with the former being the most preferred. The hydrogen

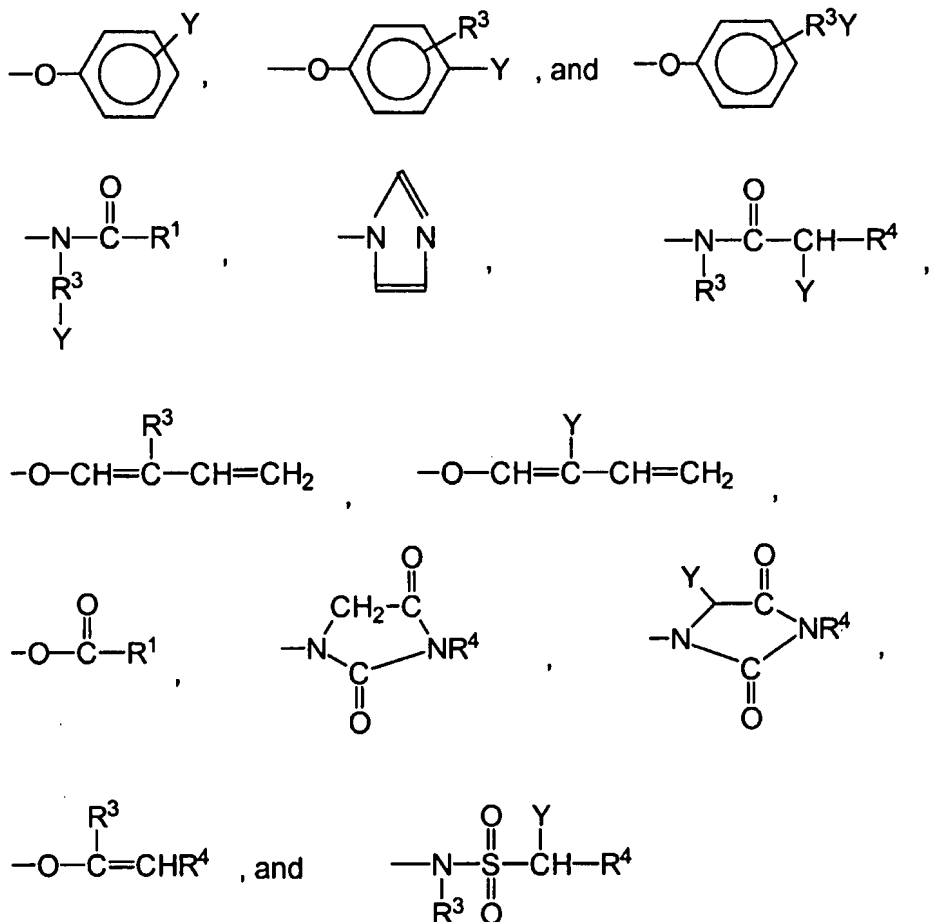
peroxide or pre-formed peracid is present in the treatment solution of the present invention at levels of from about 1 to about 50 g/L, more preferably from about 2 to about 30 g/L and most preferably from about 2 to about 20g/L. The hydrophobic activator or peracid is then employed at molar ratios of activator or peracid to peroxide of from about 1:1 to about 1:50, more preferably from about 1:2 to about 1:30 and even more preferably from about 1:5 to about 1:15. Meanwhile, the ratio of bleaching solution or liquor to the amount of fabric is from about 5:1 to 100:1, more preferably from about 5:1 to about 40:1 and most preferably from about 8:1 to about 20:1 for batch processing. For continuous process or pad-steam process the liquor pick-up will be from about 40% to about 200% on weight of fabric, preferably from about 50 to about 150% on weight of fabric.

Particularly useful and preferred is the combination of hydrogen peroxide and hydrophobic bleach activators, and in particular the alkanoyloxy class of bleach activators having the general formula:



wherein R is an alkyl chain having from about 5 to about 17, preferably from about 7 to about 11 carbon atoms and L can be essentially any suitable leaving group. A leaving group is any group that is displaced from the bleaching activator as a consequence of the nucleophilic attack on the bleach activator by the perhydroxide anion. This, the perhydrolysis reaction, results in the formation of the peroxycarboxylic acid. Generally, for a group to be a suitable leaving group it must exert an electron attracting effect. It should also form a stable entity so that the rate of the back reaction is negligible. This facilitates the nucleophilic attack by the perhydroxide anion.

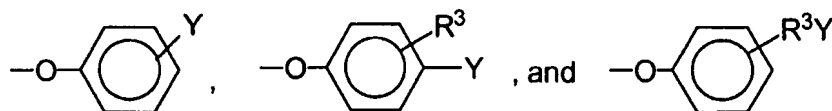
The L group must be sufficiently reactive for the reaction to occur within the optimum time frame (e.g., a wash cycle). However, if L is too reactive, this activator will be difficult to stabilize for use in a bleaching composition. These characteristics are generally paralleled by the pKa of the conjugate acid of the leaving group, although exceptions to this convention are known. Ordinarily, leaving groups that exhibit such behavior are those in which their conjugate acid has a pKa in the range of from about 4 to about 13, preferably from about 6 to about 11 and most preferably from about 8 to about 11. For the purposes of the present invention, L is selected from the group consisting of:



5 and mixtures thereof, wherein R<sup>1</sup> is an alkyl, aryl, or alkaryl group containing from about 1 to about 14 carbon atoms, R<sup>3</sup> is an alkyl chain containing from 1 to about 8 carbon atoms, R<sup>4</sup> is H or R<sup>3</sup>, and Y is H or a solubilizing group.

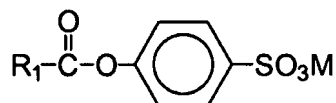
The preferred solubilizing groups are -SO<sub>3</sub><sup>-</sup>M<sup>+</sup>, -CO<sub>2</sub><sup>-</sup>M<sup>+</sup>, -SO<sub>4</sub><sup>-</sup>M<sup>+</sup>, -N<sup>+</sup>(R<sup>3</sup>)<sub>4</sub>X<sup>-</sup> and O<-N(R<sup>3</sup>)<sub>3</sub> and most preferably -SO<sub>3</sub><sup>-</sup>M<sup>+</sup> and -CO<sub>2</sub><sup>-</sup>M<sup>+</sup> wherein R<sup>3</sup> is an alkyl chain  
 10 containing from about 1 to about 4 carbon atoms, M is a cation which provides solubility to the bleach activator and X is an anion which provides solubility to the bleach activator. Preferably, M is an alkali metal, ammonium or substituted ammonium cation, with sodium and potassium being most preferred, and X is a halide, hydroxide, methylsulfate or acetate anion. It should be noted that bleach activators with a leaving group that does not contain a solubilizing groups  
 15 should be well dispersed in the bleaching solution in order to assist in their dissolution.

Preferred bleach activators are those of the above general formula wherein L is selected from the group consisting of:



wherein  $R^3$  is as defined above and Y is  $-\text{SO}_3^- \text{M}^+$  or  $-\text{CO}_2^- \text{M}^+$  wherein M is as defined above.

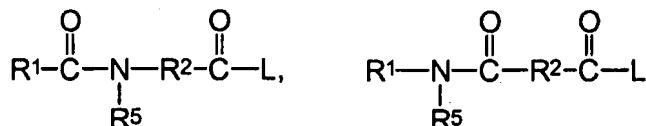
Most preferred among the bleach activators of use in the present invention, are alkanoyloxybenzenesulfonates of the formula:



wherein  $R_1$  contains from about 7 to about 12, preferably from about 8 to about 11, carbon atoms and M is a suitable cation, such as an alkali metal, ammonium, or substituted ammonium cation, with sodium and potassium being most preferred.

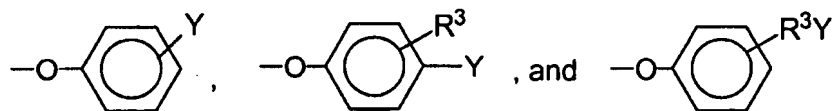
Highly preferred hydrophobic alkanoyloxybenzenesulfonates are selected from the group consisting of nonanoyloxybenzenesulfonate, 3,5,5-trimethylhexanoyloxybenzene-sulfonate, 2-ethylhexanoyloxybenzenesulfonate, octanoyloxybenzenesulfonate, decanoyloxybenzenesulfonate, dodecanoyloxybenzenesulfonate, and mixtures thereof.

Alternatively, amido derived bleach activators may be employed in the present invention. These activators are amide substituted compounds of the general formulas:



or mixtures thereof, wherein  $R^1$  is an alkyl, aryl, or alkaryl group containing from about 1 to about 14 carbon atoms,  $R^2$  is an alkylene, arylene or alkarylene group containing from about 1 to about 14 carbon atoms,  $R^5$  is H or an alkyl, aryl, or alkaryl group containing from about 1 to about 10 carbon atoms and L is a leaving group as defined above.

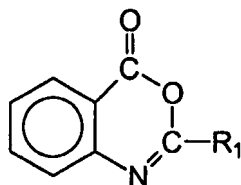
Preferred bleach activators are those of the above general formula wherein  $R^1$  is an alkyl group containing from about 6 to about 12 carbon atoms,  $R^2$  contains from about 1 to about 8 carbon atoms, and  $R^5$  is H or methyl. Particularly preferred bleach activators are those of the above general formulas wherein  $R^1$  is an alkyl group containing from about 7 to about 10 carbon atoms and  $R^2$  contains from about 4 to about 5 carbon atoms and wherein L is selected from the group consisting of:



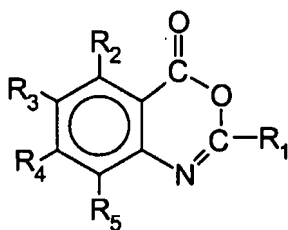
wherein  $R^3$  is as defined above and Y is  $-\text{SO}_3^- \text{M}^+$  or  $-\text{CO}_2^- \text{M}^+$  wherein M is as defined above.

Another important class of bleach activators provide organic peracids as described herein by ring-opening as a consequence of the nucleophilic attack on the carbonyl carbon of the cyclic ring by the perhydroxide anion. For instance, this ring-opening reaction in caprolactam  
 5    cyclic ring by the perhydroxide anion. For instance, this ring-opening reaction in caprolactam  
 activators involves attack at the caprolactam ring carbonyl by hydrogen peroxide or its anion. Since attack of an acyl caprolactam by hydrogen peroxide or its anion occurs preferably at the  
 exocyclic carbonyl, obtaining a significant fraction of ring-opening may require a catalyst. Another example of ring-opening bleach activators can be found in the benzoxazin type  
 10    activators.

Such activator compounds of the benzoxazin-type, have the formula:

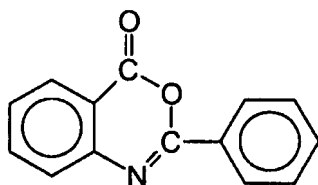


including the substituted benzoxazins of the type



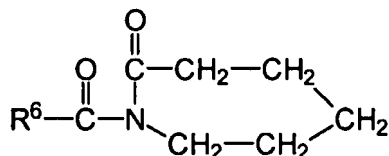
15    wherein  $R_1$  is H, alkyl, alkaryl, aryl, arylalkyl, and wherein  $R_2$ ,  $R_3$ ,  $R_4$ , and  $R_5$  may be the same or different substituents selected from H, halogen, alkyl, alkenyl, aryl, hydroxyl, alkoxyl, amino, alkyl amino,  $\text{COOR}_6$  (wherein  $R_6$  is H or an alkyl group) and carbonyl functions.

A preferred activator of the benzoxazin-type is:



When the activators are used, optimum surface bleaching performance is obtained with washing solutions wherein the pH of such solution is between about 8.5 and 10.5 and preferably between 9.5 and 10.5 in order to facilitate the perhydrolysis reaction.

N-acyl caprolactam bleach activators may be employed in the present invention. These  
5 activators have the formula:



wherein R<sup>6</sup> is H or an alkyl, aryl, alkoxyaryl, or alkaryl group containing from 1 to 12 carbons. Caprolactam activators wherein the R<sup>6</sup> moiety contains at least about 6, preferably from 6 to about 12, carbon atoms provide hydrophobic bleaching which affords nucleophilic and body  
10 soil clean-up, as noted above.

Highly preferred hydrophobic N-acyl caprolactams are selected from the group consisting of benzoyl caprolactam, octanoyl caprolactam, nonanoyl caprolactam, decanoyl caprolactam, undecenoyl caprolactam, 3,5,5-trimethylhexanoyl caprolactam, and mixtures thereof.

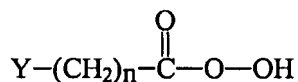
Alternatively, a pre-formed peracid may be employed in lieu of the peroxide and  
 15 activator. The pre-formed hydrophobic peracid are preferably selected from the group consisting  
 of percarboxylic acids and salts, percarbonic acids and salts, perimidic acids and salts,  
 peroxymonosulfuric acids and salts, and mixtures thereof. examples of which are described in  
 U.S. Patent No. 5,576,282 to Miracle et al.

One class of suitable organic peroxydicarboxylic acids have the general formula:

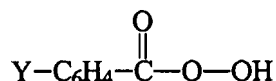


wherein R is an alkylene or substituted alkylene group containing from 1 to about 22 carbon atoms or a phenylene or substituted phenylene group, and Y is hydrogen, halogen, alkyl, aryl, -C(O)OH or -C(O)OOH.

Organic peroxyacids suitable for use in the present invention can contain either one or  
25 two peroxy groups and can be either aliphatic or aromatic. When the organic peroxycarboxylic  
acid is aliphatic, the unsubstituted peracid has the general formula:



where Y can be, for example, H, CH<sub>3</sub>, CH<sub>2</sub>Cl, C(O)OH, or C(O)OOH; and n is an integer from 0 to 20. When the organic peroxycarboxylic acid is aromatic, the unsubstituted peracid has the general formula:



5 wherein Y can be, for example, hydrogen, alkyl, alkylhalogen, halogen, C(O)OH or C(O)OOH.

Typical monoperoxy acids useful herein include alkyl and aryl peroxyacids such as:

- (i) peroxybenzoic acid and ring-substituted peroxybenzoic acid, e.g. peroxy-*a*-naphthoic acid, monoperoxyphthalic acid (magnesium salt hexahydrate), and *o*-carboxybenzamidoperoxyhexanoic acid (sodium salt);
- 10 (ii) aliphatic, substituted aliphatic and arylalkyl monoperoxy acids, e.g. peroxyauric acid, peroxysebacic acid, N-nonylaminoperoxycaproic acid (NAPCA), N,N-(3-octylsuccinoyl)aminoperoxycaproic acid (SAPA) and N,N-phthaloylaminoperoxycaproic acid (PAP);
- (iii) amidoperoxyacids, e.g. monononylamide of either peroxysebacic acid (NAPSA)
- 15 or of peroxyadipic acid (NAPAA).

Typical diperoxyacids useful herein include alkyl diperoxyacids and aryldiperoxyacids, such as:

- (iv) 1,12-diperoxydodecanedioic acid;
- (v) 1,9-diperoxyazelaic acid;
- 20 (vi) diperoxysebacic acid; diperoxysebacic acid and diperoxyisophthalic acid;
- (vii) 2-decyldiperoxybutane-1,4-dioic acid;
- (viii) 4,4'-sulfonylbis(2-peroxybenzoic acid).

Such bleaching agents are disclosed in U.S. Patent 4,483,781, Hartman, issued November 20, 1984, U.S. Patent 4,634,551 to Burns et al., European Patent Application 0,133,354, Banks et al. published February 20, 1985, and U.S. Patent 4,412,934, Chung et al. issued November 1, 1983. Sources also include 6-nonylamino-6-oxoperoxycaproic acid as fully described in U.S. Patent 4,634,551, issued January 6, 1987 to Burns et al. Persulfate compounds such as for example OXONE, manufactured commercially by E.I. DuPont de Nemours of Wilmington, DE can also be employed as a suitable source of peroxydisulfuric acid.

30 In addition to the hydrophobic bleaching ingredients described, the treatment bath or liquor of the present invention further includes a de-sizing system. De-sizing involves the removal of sizing agents such as starch and polyvinyl alcohol added to the yarns prior to weaving. The de-

sizing system involves the use of an aqueous solution of de-sizing agents such as amylase enzymes, alkali agents and oxidative agents all of which are well known in the art and well within the level of ordinary skill in the art. The preferred de-sizing system involves the use of amylase enzymes as are known in the art. The de-sizing agents are employed at levels of from about 0.5 to  
5 about 20g/l, more preferably from about 0.5 to about 10 g/l, and most preferably from about 1 to about 5 g/l.

The treatment solutions of the present invention may also include various adjunct ingredients. Such ingredients include sequestering or chelating agents, wetting agents, pH control agents, bleach catalysts, stabilizing agents, detergents and mixtures thereof. Wetting agents are  
10 typically selected from surfactants and in particular nonionic surfactants. When employed wetting agents are typically included at levels of from about 0.1 to about 20 g/L, more preferably from about 0.5 to about 20 g/L, and more preferably 0.5 to about 10 g/L of the bath. Stabilizing agents are employed for a variety of reasons including buffering capacity, sequestering, dispersing and in addition enhancing the performance of the surfactants. Stabilizing agents are well known  
15 with both inorganic or organic species being well known and silicates and organophosphates gaining the broadest acceptance and when present are employed at levels of from about 0.01 to about 30 g/L, more preferably from about 0.01 to about 20 g/L and most preferably from about 0.01 to about 10 g/L of the bath. In preferred optional embodiments of the present invention, sodium hydroxide is included in the treatment solution at levels of from about 1 to about 40 g/L,  
20 more preferably from about 2 to about 30 g/L and most preferably at levels of from about 2 to about 20 g/L.

Chelating agents may also be employed and can be selected from the group consisting of amino carboxylates, amino phosphonates, polyfunctionally-substituted aromatic chelating agents and mixtures therein, all as hereinafter defined.

25 Amino carboxylates useful as optional chelating agents include ethylenediaminetetraacetates, N-hydroxyethylethylenediaminetriacetates, nitrilotriacetates, ethylenediamine tetrapropionates, triethylenetetraaminehexacetates, phosphonates to not contain alkyl or alkenyl groups with more than about 6 carbon atoms.

Polyfunctionally-substituted aromatic chelating agents are also useful in the compositions  
30 herein. See U.S. Patent 3,812,044, issued May 21, 1974, to Connor et al. Preferred compounds of this type in acid form are dihydroxydisulfobenzenes such as 1,2-dihydroxy-3,5-disulfobenzenediethylenetriaminepentaacetates, and ethanoldiglycines, alkali metal, ammonium, and substituted ammonium salts therein and mixtures therein.



Amino phosphonates are also suitable for use as chelating agents in the compositions of the invention when at least low levels of total phosphorus are permitted.

A preferred biodegradable chelator for use herein is ethylenediamine disuccinate ("EDDS"), especially the [S,S] isomer as described in U.S. Patent 4,704,233, November 3, 1987,  
 5 to Hartman and Perkins.

When present, chelating agents are employed at levels of from about 0.01 to about 10 g/L, more preferably from about 0.1 to about 5 g/L, and most preferably from about 0.2 to about 2 g/L.

Bleach catalysts may also be employed in the treatment solutions of the present invention. One type of metal-containing bleach catalyst is a catalyst system comprising a transition metal  
 10 cation of defined bleach catalytic activity, such as copper, iron, titanium, ruthenium tungsten, molybdenum, or manganese cations, an auxiliary metal cation having little or no bleach catalytic activity, such as zinc or aluminum cations, and a sequester having defined stability constants for the catalytic and auxiliary metal cations, particularly ethylenediaminetetraacetic acid, ethylenediaminetetra (methylenephosphonic acid) and water-soluble salts thereof. Such catalysts  
 15 are disclosed in U.S. Pat. 4,430,243.

Other types of bleach catalysts include the manganese-based complexes disclosed in U.S. Pat. 5,246,621 and U.S. Pat. 5,244,594. Preferred examples of these catalysts include  $\text{Mn}^{\text{IV}}_2(\text{u-O})_3(1,4,7\text{-trimethyl-1,4,7-triazacyclononane})_2(\text{PF}_6)_2$  ("MnTACN"),  $\text{Mn}^{\text{III}}_2(\text{u-O})_1(\text{u-OAc})_2(1,4,7\text{-trimethyl-1,4,7-triazacyclononane})_2(\text{ClO}_4)_2$ ,  $\text{Mn}^{\text{IV}}_4(\text{u-O})_6(1,4,7\text{-triazacyclononane})_4(\text{ClO}_4)_2$ ,  $\text{Mn}^{\text{III}}\text{Mn}^{\text{IV}}_4(\text{u-O})_1(\text{u-OAc})_2(1,4,7\text{-trimethyl-1,4,7-triazacyclononane})_2(\text{ClO}_4)_3$ , and mixtures thereof. See also European patent application publication no. 549,272. Other ligands suitable for use herein include 1,5,9-trimethyl-1,5,9-triazacyclododecane, 2-methyl-1,4,7-triazacyclononane, 2-methyl-1,4,7-triazacyclononane, and mixtures thereof. For examples of other suitable bleach catalysts herein see U.S. Pat. 4,246,612,  
 20 U.S. Pat. 5,227,084 and WO 95/34628, December 21, 1995, the latter relating to particular types of iron catalyst.

See also U.S. Pat. 5,194,416 which teaches mononuclear manganese (IV) complexes such as  $\text{Mn}(1,4,7\text{-trimethyl-1,4,7-triazacyclononane}(\text{OCH}_3)_3)(\text{PF}_6)$ .

Still another type of bleach catalyst, as disclosed in U.S. Pat. 5,114,606, is a water-soluble  
 30 complex of manganese (II), (III), and/or (IV) with a ligand which is a non-carboxylate polyhydroxy compound having at least three consecutive C-OH groups. Preferred ligands include sorbitol, iditol, dulcitol, mannitol, xylitol, arabitol, adonitol, meso-erythritol, meso-inositol, lactose, and mixtures thereof.

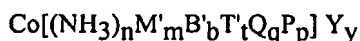
U.S. Pat. 5,114,611 teaches another useful bleach catalyst comprising a complex of transition metals, including Mn, Co, Fe, or Cu, with an non-(macro)-cyclic ligand. Preferred ligands include pyridine, pyridazine, pyrimidine, pyrazine, imidazole, pyrazole, and triazole rings. Optionally, said rings may be substituted with substituents such as alkyl, aryl, alkoxy, halide, and nitro. Particularly preferred is the ligand 2,2'-bispyridylamine. Preferred bleach catalysts include Co-, Cu-, Mn-, or Fe- bispyridylmethane and bispyridylamine complexes. Highly preferred catalysts include Co(2,2'-bispyridylamine)Cl<sub>2</sub>, Di(isothiocyanato)bispyridylamine-cobalt (II), trisdipyrldylamine-cobalt(II) perchlorate, Co(2,2-bispyridylamine)<sub>2</sub>O<sub>2</sub>ClO<sub>4</sub>, Bis-(2,2'-bispyridylamine) copper(II) perchlorate, tris(di-2-pyridylamine) iron(II) perchlorate, and mixtures thereof.

Other bleach catalyst examples include Mn gluconate, Mn(CF<sub>3</sub>SO<sub>3</sub>)<sub>2</sub>, Co(NH<sub>3</sub>)<sub>5</sub>Cl, and the binuclear Mn complexed with tetra-N-dentate and bi-N-dentate ligands, including N<sub>4</sub>Mn<sup>III</sup>(u-O)<sub>2</sub>Mn<sup>IV</sup>N<sub>4</sub>)<sup>+</sup> and [Bipy<sub>2</sub>Mn<sup>III</sup>(u-O)<sub>2</sub>Mn<sup>IV</sup>bipy<sub>2</sub>](ClO<sub>4</sub>)<sub>3</sub>.

Particularly preferred manganese catalyst for use herein are those which are fully disclosed in WO 98/23249, WO 98/39098, WO 98/39406 and WO 98/39405, the disclosures of which, are herein incorporated by reference.

Other bleach catalysts are described, for example, in European patent application, publication no. 408,131 (cobalt complex catalysts), European patent applications, publication nos. 384,503, and 306,089 (metallo-porphyrin catalysts), U.S. 4,728,455 (manganese/multidentate ligand catalyst), U.S. 4,711,748 and European patent application, publication no. 224,952, (absorbed manganese on aluminosilicate catalyst), U.S. 4,601,845 (aluminosilicate support with manganese and zinc or magnesium salt), U.S. 4,626,373 (manganese/ligand catalyst), U.S. 4,119,557 (ferric complex catalyst), German Pat. specification 2,054,019 (cobalt chelant catalyst) Canadian 866,191 (transition metal-containing salts), U.S. 4,430,243 (chelants with manganese cations and non-catalytic metal cations), and U.S. 4,728,455 (manganese gluconate catalysts).

Preferred are cobalt (III) catalysts having the formula:



wherein cobalt is in the +3 oxidation state; n is an integer from 0 to 5 (preferably 4 or 5; most preferably 5); M' represents a monodentate ligand; m is an integer from 0 to 5 (preferably 1 or 2; most preferably 1); B' represents a bidentate ligand; b is an integer from 0 to 2; T' represents a tridentate ligand; t is 0 or 1; Q is a tetradentate ligand; q is 0 or 1; P is a pentadentate ligand; p is 0 or 1; and n + m + 2b + 3t + 4q + 5p = 6; Y is one or more appropriately selected counteranions present in a number y, where y is an integer from 1 to 3 (preferably 2 to 3; most preferably 2 when Y is a -1 charged anion), to obtain a charge-balanced salt, preferred Y are selected from the

group consisting of chloride, nitrate, nitrite, sulfate, citrate, acetate, carbonate, and combinations thereof; and wherein further at least one of the coordination sites attached to the cobalt is labile under automatic dishwashing use conditions and the remaining coordination sites stabilize the cobalt under automatic dishwashing conditions such that the reduction potential for cobalt (III) to cobalt (II) under alkaline conditions is less than about 0.4 volts (preferably less than about 0.2 volts) versus a normal hydrogen electrode. Some preferred catalysts are the chloride salts having the formula  $[\text{Co}(\text{NH}_3)_5\text{Cl}] Y_y$ , and especially  $[\text{Co}(\text{NH}_3)_5\text{Cl}]\text{Cl}_2$ .

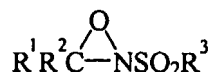
More preferred are the present invention compositions which utilize cobalt (III) bleach catalysts having the formula:



wherein cobalt is in the +3 oxidation state; n is 4 or 5 (preferably 5); M is one or more ligands coordinated to the cobalt by one site; m is 0, 1 or 2 (preferably 1); B is a ligand coordinated to the cobalt by two sites; b is 0 or 1 (preferably 0), and when b=0, then m+n = 6, and when b=1, then m=0 and n=4; and T is one or more appropriately selected counteranions present in a number y, where y is an integer to obtain a charge-balanced salt (preferably y is 1 to 3; most preferably 2 when T is a -1 charged anion); and wherein further said catalyst has a base hydrolysis rate constant of less than  $0.23 \text{ M}^{-1} \text{ s}^{-1}$  (25°C). These materials are more fully disclosed in U.S. Patent Nos. 5,559,261, 5,597,936, 5,705,464, 5,703,030 and 5,962,386 the disclosures of which are herein incorporated by reference.

As a practical matter, and not by way of limitation, the solutions herein can be adjusted to provide on the order of at least one part per hundred million of the active bleach catalyst species in the aqueous medium, and will preferably provide from about 0.01 ppm to about 25 ppm, more preferably from about 0.05 ppm to about 10 ppm, and most preferably from about 0.1 ppm to about 5 ppm, of the bleach catalyst species in the liquor.

Also useful herein are any of the known organic bleach catalysts, oxygen transfer agents or precursors therefor. These include the compounds themselves and/or their precursors, for example any suitable ketone for production of dioxiranes and/or any of the hetero-atom containing analogs of dioxirane precursors or dioxiranes, such as sulfonimines  $\text{R}^1\text{R}^2\text{C}=\text{NSO}_2\text{R}^3$ , see EP 446 982 A, published 1991 and sulfonyloxaziridines, for example:



see EP 446,981 A, published 1991. Preferred examples of such materials include hydrophilic or hydrophobic ketones, used especially in conjunction with monoperoxysulfates to produce

dioxiranes in situ, and/or the imines described in U.S. 5,576,282 and references described therein. Oxygen bleaches preferably used in conjunction with such oxygen transfer agents or precursors include percarboxylic acids and salts, percarbonic acids and salts, peroxymonosulfuric acid and salts, and mixtures thereof. See also U.S. 5,360,568; U.S. 5,360,569; and U.S. 5,370,826. In a  
5 highly preferred embodiment, the invention relates to a detergent composition which incorporates a transition-metal bleach catalyst in accordance with the invention, and organic bleach catalyst such as one named hereinabove.

Additional components that may be a part of the solution of the present invention include from about 1.0% to about 4.0% by weight of the agent of an alkali salt, such as sodium  
10 bicarbonate in a preferred amount of about 2.0% by weight. From about 0.25% to about 2.0% by weight of the agent of a de-foamer, such as polydimethyl siloxane may also be included. Other optional components include for example, lubricants and softeners.

One of ordinary skill in the art will recognize that the process conditions to be used in performing the present invention may be selected so as to match a particular equipment or a  
15 particular type of process which it is desirable to use. For example, while the textile components preferably remain in contact with the treatment solution at a temperature of from about 20 to about 80 °C, preferably from about 50°C to about 80 °C and for a period of time suitable for treating the textile component which is at least about 15 minutes more preferably from about 30 minutes to about 180 minutes, preferably from about 30 to about 120 minutes and most preferably  
20 from about 30 to about 90 minutes. Of course, one of ordinary skill in the art will recognize that the reaction conditions such as time and temperature will vary depending upon the equipment employed and the fabrics treated.

Preferred examples of process types to be used in connection with the present invention include Jigger/Winch, Pad-Roll and Pad-Steam types. These types are dealt with in further detail  
25 below. The combined process of the invention may be carried out as a batch, semi-continuous or continuous process using steam or the principles of cold-bleaching. As an example the process may comprise the following steps:

a) impregnating the fabric in a de-sizing and bleaching bath as described herein followed by squeezing out excessive liquid so as to maintain the quantity of liquor necessary for the  
30 reaction to be carried out (normally between 60 and 120% of the weight of the dry fabric), (b) subjecting the impregnated fabric to steaming so as to bring the fabric to the desired reaction temperature, generally between about 20 and about 80 °C, and (c) holding by rolling up or

pleating the cloth in a J-Box, U-Box, carpet machine or the like for a sufficient period of time to allow the de-sizing and bleaching to occur.

As mentioned above, scouring may be an inherent result obtained when performing the combined process of the invention. However, for certain types of fabric it may be advantageous  
5 and/or necessary to subject the fabric to a scouring treatment in order to obtain a final product of a desired quality. In such cases, the present invention may be employed as a combined de-sizing, bleaching and scouring process.

The method of the present invention involves providing a non-finished textile component into the treatment solution as described. The textile component may comprise fibers, yarns and  
10 fabrics including wovens, nonwovens and knits. By non-finished, it is intended that the textile component be a material that has not been dyed, printed, or otherwise provided a finishing step such as durable press. Of course, one of ordinary skill in the art will recognize that the textile component of the present invention are those that have not been passed through a garment or other manufacturing process involving cutting and sewing of the material.

15 The present process may be employed with most any natural material including cellulose such as cotton, linen and regenerated cellulose such as rayon and lyocell. Both 100% natural fibers, yarns and fabrics may be employed or blends with synthetic materials may be employed as well. For the purposes of the present invention, natural fibers may include cellulose as described herein, wool both pure and blends, silks, sisal, flax and jute.

20 The method of the present invention may include the further steps of singeing, scouring, and mercerization in conjunction with the treatment step. While scouring may be employed in a separate step, in preferred embodiments the scouring step is included in the one step treatment of the present invention via the inclusion of sodium hydroxide in the treatment bath thereby combining, bleaching, de-sizing and scouring into a single step. When doing so, sodium  
25 hydroxide is included in levels of from about 1 to about 40 g/l, more preferably from about 2 to about 30 g/l and more preferably from about 2 to about 20 g/l.

The steps of singeing and mercerization, and in less preferred embodiments scouring, may be performed in various combinations and orders and one of ordinary skill in the art will recognize that varying combinations are possible. The scouring step of the present invention  
30 involves the removal of natural or synthetic impurities from the textiles such as waxes and oils. Optional ingredients in the alkaline bath include wetting agents and chelating agents.

The mercerization step of the present invention involves the application of high concentrations of alkali such as sodium hydroxide in conjunction with stretching and pulling of

the textiles to restore fiber strength and improve luster while singeing involves passing the textiles over an open flame to remove loose fibers or strands. Scouring, mercerization and singeing are well known to one of ordinary skill in the art and will be well recognized and within the level of skill of the artisan.

5 Of course the process of the present invention includes in the preferred application a washing step or series of washing steps following the method of the present invention. Washing of treated textiles is well known and within the level of skill of the artisan. Washing stages will be typically present after each of the scouring and mercerization step when present as well as after the treatment step of the present invention. In addition, the treatment step and optional scouring  
10 and mercerization steps of the present invention when present may in preferred embodiments include a wet-out or pre-wetting step to ensure even or uniform wettness in the textile component.

The method of the present invention provides superior wettability to textile components treated via the method. Wettability of the textiles is important to any dyeing and finishing of the textiles. Wettability leads to superior penetration of the textile by the dye or finish agents and a  
15 superior dye and/or finishing result. Accordingly, the wettability of the textile is an indication of how effective the treatment process has been. Higher wettability means a more effective and superior treatment process, i.e., a shorter period of time for wetting. Conventional textile peroxygen bleaching has provided acceptable wetting profiles only at temperature in excess of 95°C while lower temperature bleaching (70°C) results in wettability profiles more than about  
20 40%. However, the process of the present invention provides fabrics that have a increase in the wettability index of less than about 10% preferably less than about 5% where the wettability index is defined as:

$$\frac{(\text{wettability at } 70^{\circ}\text{C}) - (\text{wettability at } 95^{\circ}\text{C})}{(\text{wettability at } 95^{\circ}\text{C})}$$

25 in percent.

For purposes of the present invention, fiber damage based on fluidity is measured via AATCC test method 82-1996 involving the dispersion of the fibers in cupriethylene diamine (CP). A representative sample of fibers of about 1.5 mm is cut and dissolved in CP as defined by the equation  $CP=120 \times \text{sample weight} \times 0.98$  in a specimen bottle with several glass balls, placed  
30 under nitrogen and dissolved by shaking for approximately 2 hours. Additional CP is added as defined by the equation  $CP=80 \times \text{sample weight} \times 0.98$  and additional shaking under nitrogen for three hours. The solution is placed under constant stirring to prevent separation of the dispersion. The solution is then measured in a calibrated Oswald Canon Fenske viscometer in a constant temperature bath of 25°C to determine the efflux time. Fluidity is then calculated from the

formula  $F=100/ctd$ , where  $c$  = viscometer constant,  $t$  = efflux time and  $d$  = density of the solution 1.052.

The following non-limiting examples further illustrate the present invention.

## 5 EXAMPLE I

The one step textile treatment of the present invention may be exemplified via the following procedure. The one step treatment was performed in an automatic lab dyeing machine with an infrared heating system (Labmat) made by the Mathis Co. The treatment bath was prepared via the addition of the ingredient specified in Table I by addition to deionized water.

10 The addition sequence was as follows: Wetting agent - Chelating agent - Stabilizer - Enzyme - Activator -  $H_2O_2$  - NaOH - Fabric. The fabric was a greige 100% cotton print cloth, plain woven with  $122\text{ g/m}^2$  before de-sizing and scouring. The original fabric whiteness was 21.74. With a liquor/fabric ratio of 15:1, the fabric is put into the treatment solution. The solution is heated from about  $20^\circ\text{C}$  to required bleaching temperature as specified in Table I in about 10 minutes,

15 maintained at the required temperature for 60 minutes, and then cooled to the room temperature. The fabric is then removed from the bleaching solution and washed with hot water and warm water to ensure that no residual alkali remains on the fabric. The fabric is then dried and conditioned under  $70^\circ\text{F}$  and 65% relative humidity for wetting and whiteness measurements. Miniscan XE Plus made by HunterLab is used to measure CIE Whiteness Index. An Instron is

20 used to evaluate the fabric strength by following the method ASTM D 5035. The fabric wettability is measured by AATCC Test Method 79-1995. The less wetting time, the better is the fabric wettability or absorbency. The results are reported in Table I.

	A	B	C
Temp ( $^\circ\text{C}$ )	70	70	70
NaOH (g/l)	3.5	3.5	3.5
$H_2O_2$ (g/l)	2.5	2.5	2.5
Activator	None	Hydrophobic <sup>1</sup>	Hydrophilic <sup>2</sup>
Molar Ratio (Activator/ $H_2O_2$ )	NA	1:10	1:10
Desizing Enzyme <sup>3</sup> (g/l)	5	5	5
Stabilizer (g/l)	1	1	1
Wetting Agent <sup>4</sup> (g/l)	2.0	2.0	2.0
Chelating Agent <sup>5</sup> (g/l)	0.5	0.5	0.5

Time (minutes)	60	60	60
Liquor/Fabric Ratio	15:1	15:1	15:1
Wettability (seconds)	17.20	1.32	11.79
CIE Whiteness	61.41	76.98	65.33

<sup>1</sup> nonanoyloxybenzene sulfonate, sodium salt, NOBS.

<sup>2</sup> tetra acetyl ethylene diamine, TAED.

<sup>3</sup> Amzyme 60 from American Emulsions

5 <sup>4</sup> Silvatrol SL from Ciba Geigy.

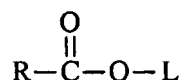
<sup>5</sup> Invatek DTPA-41 from Ciba Geigy



**WHAT IS CLAIMED IS:**

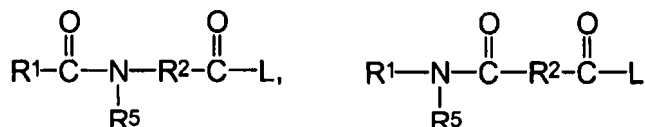
1. A method for the one step preparation of a non-finished textile component characterized by the steps of providing a non-finished textile component, saturating said textile component with an aqueous treatment solution characterized by a hydrophobic bleaching system and a enzymatic de-sizing system and allowing said treatment solution to remain in contact with said textile component for a period of time sufficient to treat said textile component.
2. The method as claimed in Claim 1 wherein the resultant treated textile component has a whiteness value on the CIE index of at least 70.
3. The method as claimed in any of Claims 1-2 wherein said hydrophobic bleaching system in said aqueous treatment solution is characterized by hydrogen peroxide and a hydrophobic bleach activator selected from the group consisting of:

a) a bleach activator of the general formula:



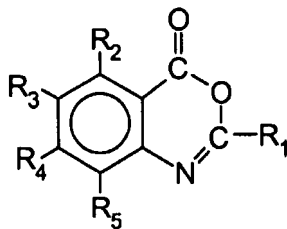
wherein R is an alkyl chain having from 5 to 17 carbon atoms and L is a leaving group;

b) a bleach activator of the general formula:



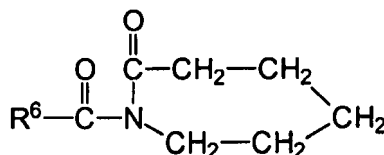
or mixtures thereof, wherein  $\text{R}^1$  is an alkyl, aryl, or alkaryl group containing from 1 to 14 carbon atoms,  $\text{R}^2$  is an alkylene, arylene or alkarylene group containing from 1 to 14 carbon atoms,  $\text{R}^5$  is H or an alkyl, aryl, or alkaryl group containing from 1 to 10 carbon atoms, and L is a leaving group;

c) a benzoxazin-type bleach activator of the formula:



wherein  $R_1$  is H, alkyl, alkaryl, aryl, arylalkyl, and wherein  $R_2$ ,  $R_3$ ,  $R_4$ , and  $R_5$  may be the same or different substituents selected from H, halogen, alkyl, alkenyl, aryl, hydroxyl, alkoxy, amino, alkylamino,  $-\text{COOR}_6$ , wherein  $R_6$  is H or an alkyl group and carbonyl functions;

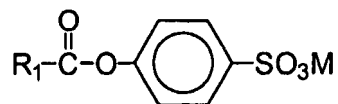
d) a N-acyl caprolactam bleach activator of the formula:



wherein  $R^6$  is H or an alkyl, aryl, alkoxyaryl, or alkaryl group containing from 1 to 12 carbons; and

e) mixtures of a, b, c and d.

4. The method as claimed in any of Claims 1-3 wherein said bleach activator is an alkanoyloxybenzenesulfonates of the formula:



wherein  $R_1$  is an alkyl group having from 8 to 11 carbon atoms and M is a suitable cation.

5. The method as claimed in any of Claims 1-4 wherein said treatment solution is at a temperature of from 20 to 80°C.
6. The method as claimed in any of Claims 1-5 wherein said treated textile component experiences a fabric strength reduction of less than 10% during said method.
7. The method as claimed in any of Claims 1-6 wherein said treated textile component experiences a fluidity increase of less than 25%.

8. A one step method for the batch preparation of a woven textile fabric characterized by the steps of:
  - a) providing an incoming non-finished woven fabric;
  - b) passing said fabric to an aqueous treatment solution, said treatment solution characterized by a mixture of a hydrophobic bleaching system and an enzymatic de-sizing system, heating said treatment solution to a temperature of from 20° to 80°C and allowing said treatment solution to contact said fabric for a period of time of from 15 to 180 minutes to treat said fabric and wherein said resultant fabric has a whiteness on said CIE scale of at least 70.
9. A method for the one step preparation of a non-finished textile component characterized by the steps of providing a non-finished textile component, saturating said textile component with an aqueous treatment solution characterized by a) hydrophobic bleaching system of hydrogen peroxide and a hydrophobic bleach activator; b) an enzymatic de-sizing system and c) a alkaline scouring agent; and allowing said treatment solution to remain in contact with said textile component for a period of time sufficient to treat said textile component.
10. The product produced by the process of any of Claims 1-9.

## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 01/04900

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 D06L1/14 D06L3/00 D06L3/02

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 D06L C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

WPI Data, EPO-Internal, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	DENTER U ET AL: "PRESSIGSAEURE IN DER TEXTILVEREDLUNG" TEXTILVEREDLUNG,CH,THURGAUER TAGBLATT, WEINFELDEN, vol. 31, no. 3/04, 1 March 1996 (1996-03-01), pages 50-55, XP000581041 ISSN: 0040-5310 abstract --- -/--	1-3,5, 8-10



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

## \* Special categories of cited documents:

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
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- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

\*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

\*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

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Date of the actual completion of the international search

22 June 2001

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## INTERNATIONAL SEARCH REPORT

International Application No.

PCT/US 01/04900

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
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A	PATENT ABSTRACTS OF JAPAN vol. 1998, no. 09, 31 July 1998 (1998-07-31) & JP 10 095996 A (KAO CORP), 14 April 1998 (1998-04-14) abstract -----	1,3-5,8, 9

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